National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2584

Trace Elements in Indoor Dust
Nominal 1 % Lead

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead and other trace elements in dust. SRM 2584 is composed of dust collected from vacuum cleaner bags used in the cleaning of interior dwelling spaces. A unit consists of 8 g of particulate material, 99+ % of which passes a 100 μm (No. 145) sieve.

Certified Values and Uncertainties: The certified values for five elements in SRM 2584 are listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values are based on measurements from two or more independent analytical methods or a single NIST primary method. Analytical methods used for the characterization of this SRM are given in Table 4. All values are reported as mass fractions [1], on a dry basis (see Instructions for Drying) and are based on measurements using a sample mass of at least 100 mg.

Reference Values and Uncertainties: Reference values for mass fractions of 10 elements are given in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. The reference values and uncertainties are based on measurements from two or more analytical methods performed at NIST and/or the U.S. Geological Survey (USGS).

Information Concentration Values: Information values are provided in Table 3 for the mass fractions of 22 additional elements. Information values are considered to be values that will be of interest and use to the SRM user, but for which insufficient information is available to assess the uncertainties associated with the values. The information values are based on measurements from a single analytical method.

Expiration of Certification: The certification of this SRM is valid within the measurement uncertainties specified until **31 December 2010**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use section). However, the certification will be nullified if the SRM is contaminated or otherwise modified.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by P.A. Pella and G.C. Turk of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh and K.R. Eberhardt of the NIST Statistical Engineering Division.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (USEPA) under the direction of project managers S.L. Harper and M.E. Beard of the EPA Office of Research and Development, National Exposure Research Laboratory, Research Triangle Park, NC.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 15 December 1999

Thomas E. Gills, Director
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NOTICE AND WARNING TO USERS

Stability: This material is considered to be stable. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

Use: To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum sample mass of 100 mg should be used and the sample should be handled according to the Instructions for Drying. Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. This SRM must be stored in an air conditioned or similar cool and dry environment away from sunlight and fumes.

Instructions for Drying: When nonvolatile elements such as cadmium, chromium, and lead are to be determined, samples should be oven dried for 2 h at 110 °C. Volatile elements, such as arsenic and mercury, should be determined on samples as-received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections should then be made to measurement values before comparing them to the certified values.

Table 1. Certified Mass Fractions

Element	Mass Fraction, in mg/kg
Arsenic	17.4 ± 4.2
Cadmium	10.0 ± 1.1
Chromium	135.0 ± 9.1
Lead	9761 ± 67
Mercury	5.20 ± 0.24

The certified values for lead and cadmium were determined by isotope dilution mass spectrometry (IDMS). The certified values for the remaining elements were determined by combining data from two or more independent analytical methods in the manner described by Schiller and Eberhardt [2]. Because of evidence of inhomogeneity, the uncertainties for arsenic, cadmium, and lead are each based on a 95 % prediction interval for the true value. This interval includes the combined effects of uncertainty components associated with material inhomogeneity, measurement uncertainty, and an allowance for differences between the analytical methods used [3]. The uncertainties for chromium and mercury, which exhibited no evidence of inhomogeneity, are each based on a 95 % confidence interval for the true value, including the combined effects of uncertainty components associated with measurement uncertainty and an allowance for differences between the analytical methods used.

Table 2. Reference Mass Fractions

Element	Mass Fraction, in mg/kg
Aluminum	23 200 ± 600
Calcium	63 300 ± 3 000
Iron	16 400 ± 1 200
Potassium	9 500 ± 1 400
Lanthanum	19 ± 2
Magnesium	15 900 ± 300
Sodium	27 700 ± 1 200
Phosphorus	2 000 ± 120
Titanium	4 200 ± 300
Zinc	2 580 ± 150

The uncertainties are based on a 95 % confidence interval for the true value, including the combined effect of the measurement uncertainty for each method and an allowance for differences between the analytical methods used [2].

Table 3. Information Mass Fractions

Element	Mass Fraction, mg/kg	Element	Mass Fraction, mg/kg
Antimony	14	Nickel	90
Barium	1300	Niobium	10
Beryllium	0.7	Rubidium	33
Bismuth	9	Scandium	4
Cerium	35	Selenium	2
Cesium	1.4	Silicon	106 000
Cobalt	10	Strontium	160
Copper	320	Thorium	4
Gallium	6.4	Uranium	1.6
Lithium	17	Vanadium	34
Manganese	370	Yttrium	10
Molybdenum	5.5		

COLLECTION, PREPARATION, AND ANALYSIS

Collection: Approximately 65 % of the material used for SRM 2584 was obtained from households in Montana, New Jersey, Ohio, and Wisconsin involved in lead poisoning intervention programs in which HEPA®[1] vacuum cleaners were used to remove dust and other surface debris from homes where cases of lead poisoning had occurred. This material was mixed with low level material taken from the sources used for the preparation of SRM 2583, namely routine vacuum cleaner bags from households, cleaning services, motels, and hotels from North Carolina, Maryland, Ohio, and New Jersey. The vacuum cleaner bags were collected under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency. The collection process was coordinated by E.D. Hardison and D.A. Binstock of the Research Triangle Institute, Research Triangle Park (RTI), NC, under the leadership of W.F. Gutknecht.

Preparation: From RTI the bags were labeled, boxed and sent to Neutron Products, Dickerson, MD, for radiation sterilization, and then shipped to NIST for processing. The initial screening and preparation to select suitable material were directed by P.A. Pella and performed by A.F. Marlow, C. Desai, and P. Seo of the NIST Analytical Chemistry Division. Final processing and blending was performed by D.G. Friend and C.N. Fales of the NIST Standard Reference Materials Program. The raw material from each bag was mixed and tumbled in a modified food processor using chopping blades and a compressed air jet. While still tumbling, the dust was separated from unwanted debris by vacuuming through a series of screens into a clean HEPA vacuum cleaner. The dust collected in this manner was then screened through a 90 μm stainless steel sieve using vibration and a vacuum. Processed sub-lots of approximately 5 kg each were set aside and analyzed for lead by x-ray fluorescence in order to develop a blending protocol for the target lead concentration. Selected high and low level sub-lots were blended in a cone blender and then bottled.

Analysis: Certification analyses were performed in the NIST Analytical Chemistry Division. Reference and information value analyses were performed by the USGS in Denver, CO, using inductively coupled plasma mass spectrometry and wavelength dispersive x-ray fluorescence spectrometry and by the NIST Analytical Chemistry Division using instrumental neutron activation analysis. Analytical methods used for this SRM are given in Table 4.

[1] Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 4. Methods used for the Analysis of SRM 2584[a]

CVAAS	**Hg**
HR-ICPMS	**As**, **Cr**
ICPMS (NIST)	Cd
ICPMS (USGS)	Al, Ba, Be, Bi, Ca, Ce, Co, Ca, Cu, Fe, Ga, K, La, Li, Mg, Mn, Mo, Na, Nb, Ni, P, Rb, Sb, Sc, Se, Sr, Th, Ti, U, V, Y, Zn
ID-ICPMS	Cd, Pb
ID-TIMS	**Cd**, **Pb**
INAA	**As**, **Cr**, Fe, **Hg**, La, Zn
WDXRF (USGS)	Al, Ca, Fe, K, Mg, Na, P, Si, Ti

[a]Methods used for establishment of certified values are indicated by bold-face type.

Methods

CVAAS	Cold vapor atomic absorption spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution quadrupole inductively coupled plasma mass spectrometry
ID-TIMS	Isotope dilution thermal ionization mass spectrometry
INAA	Instrumental neutron activation analysis
HR-ICPMS	High resolution inductively coupled plasma mass spectrometry
WDXRF	Wavelength dispersive x-ray fluorescence spectrometry

Analysts - NIST Analytical Chemistry Division

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User Experience with SRM 2584

In order to demonstrate user experience with SRM 2584, a number of laboratories analyzed this material, each using its typical method. For lead, this was done through the Environmental Lead Proficiency Analytical Testing Program (ELPAT), where SRM 2584 was loaded onto dust wipes and included as an unknown for ELPAT Round Number 022. The ELPAT results have been converted from µg/wipe to mg/kg. Data for arsenic, cadmium, chromium, and mercury were supplied by volunteer laboratories in a round robin exercise organized by NIST. For these elements the SRM was analyzed directly. Among the participants, the range of digestion procedures used included various standard and in house hotplate, microwave, hot block, and water bath methods. Instrumental methods included ICPMS, ICP Atomic Emission Spectrometry, Graphite Furnace AAS, Flame AAS, and CVAAS. The results from this study were not used in calculating the certified values of SRM 2584. The results are given in Table 5 below. The summary statistics are based on 118 reported results for lead and 13 to 16 results for the other elements.

Table 5. Results of Round Robin Exercise

Element	Mean, in mg/kg	Minimum, in mg/kg	Maximum, in mg/kg	s[a], in mg/kg
As	21.0	17.5	40.2	7.2
Cd	9.8	8.2	13.9	1.6
Cr	70.3	47.4	108	18.2
Hg	4.1	1.0	5.5	1.3
Pb	8953	7633	9845	602

[a] s is one standard deviation.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (1994).

[2] Schiller, S.B. and Eberhardt, K.R., "Combining Data from Independent Chemical Analysis Methods," Spectrochimica Acta, **46B**, pp. 1607-1613, (1991).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); (available at http://physics.nist.gov/Pubs/).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.